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7. Formation of Ultrafine Powders. (IV)

Formation of Barium Carbonate by the Carbonation Method

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In the electron-tube the oxide-coated cathode is used to increase the thermionic emission, and for this purpose Ca-Sr-Ba-triple carbonate is coated on the tungsten filament. Previously, one of the authors studied on the formation of ultrafine Ca- and Mg-carbonates by the carbonation method (*this Bull.* 18, 115, 117 (1940); 26, 80 (1951)). In this experiment the kinetics of the carbonation of $\text{Ba}(\text{OH})_2$ by CO_2 gas was studied and the shape and size of BaCO_3 were observed by electron microscope.

(A) **Kinetics of the Reaction.** CO_2 gas was introduced to $\text{Ba}(\text{OH})_2$ solution at constant velocity, and pH of the reactant was measured every 30 seconds. The change of the concentration of $\text{Ba}(\text{OH})_2$ was measured by the titration with 0.1 *N* HCl. In Table 1 the conditions of the formation of BaCO_3 are shown collectively.

Table 1. The condition of the reaction and the size of BaCO_3 .

Sample No.	Temperature °C	Concentration of $\text{Ba}(\text{OH})_2$ solution <i>M</i>	Concentration of CO_2 gas * %	Size of BaCO_3 precipitate** μ
1	10	0.100	100	
2	20	0.100	100	0.1~1.0
3	30	0.100	100	0.2~2.0
4	10	0.100	25	
5	10	0.224	100	
6	10	0.224	25	
7	20	0.100	25	0.1~0.7
8	20	0.265	100	0.1~1.0
9	20	0.265	25	0.5~2.0
10	30	0.352	100	2.0~9.0

* The flowing velocity was kept constant, 7.3 cc/sec.

** The width of all the crystals is 0.1 μ . Only the length is indicated here.

At an early stage of the reaction pH increased somewhat and the quantity of $\text{Ba}(\text{OH})_2$ decreased with the reaction, which was probably the first order, then pH decreased about 1 until the concentration of $\text{Ba}(\text{OH})_2$ decreased to 1/10 of the initial concentration, and pH decreased sharply during the reaction of residual $\text{Ba}(\text{OH})_2$ to 6.5 as shown in Figs. 1 and 2.

As shown in Fig. 2 the higher the temperature, the slower the reaction velocity is. CO_2 used for the reaction was 100 and 25 %, which was diluted with air. With increase of the concentration of CO_2 the reaction velocity became faster and the reaction

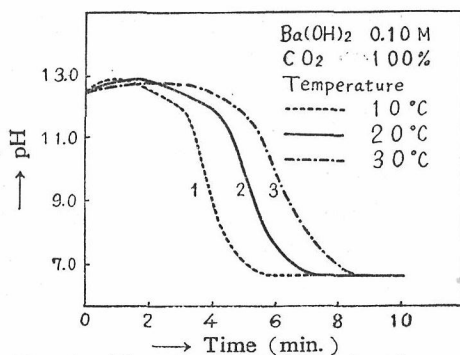


Fig. 1. The change of pH during the reaction.

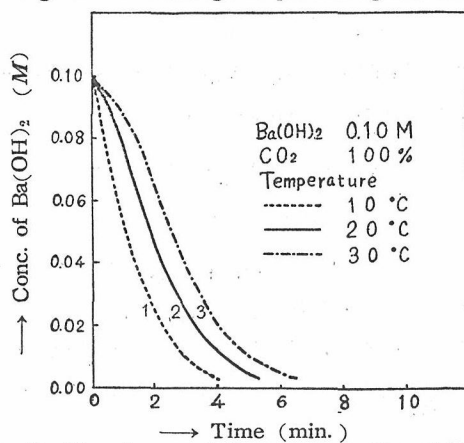


Fig. 2. The change of the concentration of Ba(OH)_2 .

time decreased. The higher the concentration of Ba(OH)_2 the slower the reaction velocity was.

It seemed that the solubility of CO_2 gas is the most dominant to the velocity of the reaction.

(B) **Morphology of Barium Carbonate Precipitates.** The precipitated BaCO_3

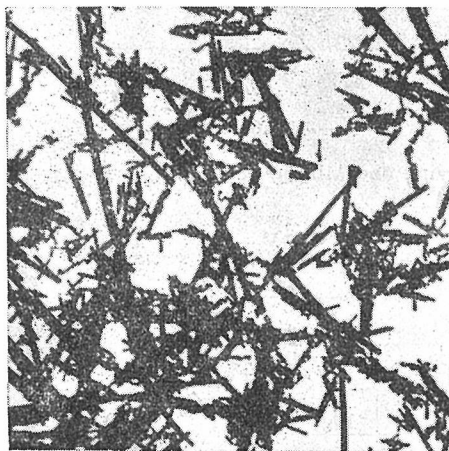


Photo 1. BaCO_3 (No.8) $\times 10,000$

was observed and photographed by electron microscope. Various shapes and sizes of CaCO_3 were formed by changing the precipitating conditions, but the shape of BaCO_3 were almost unchanged by the reaction conditions. The shape of BaCO_3 was rod and the width was constant, 0.1μ , and the length were $0.1 \sim 2.0 \mu$. One of them is shown in Photo 1. The size was scarcely changed by the reaction condition, but it was somewhat larger, when the temperature and the concentration of CO_2 were higher and the concentration of $\text{Ba}(\text{OH})_2$ was lower as shown in Table 1. No aging effect was observed for 3 months. The precipitate turned to coagulated form by filtering and drying.

8. A Method of Sampling for Obtaining the Daily Average of Batch Composition

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Introducing the statistical treatment recently developed for the analysis of coal etc. to that of glass batch the method of sampling and dividing for finding out the statistically acceptable daily average of the batch composition as well as the method of routine analysis suitable for getting the results within the practical time restriction

Steps

